



10/021,982
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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OCT 24 2003

TC 1700

In re Application of: Gyanesh P. Khare

Serial No.: 10/021,982

Group Art Unit: 1764

Filed: November 28, 2001

Examiner: James Arnold, Jr.

For: DESULFURIZATION AND NOVEL SORBENT FOR SAME

Rule 131 Declaration

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

1. I, Gyanesh P. Khare, the inventor of the above-referenced patent application, make this declaration to establish actual reduction to practice of the claimed invention in the United States on a date prior to August 25, 1999, which is the effective date of U.S. Patent No. 6,254,766, to Sughrue et al. (hereinafter the "Sughrue '766 Patent") under 35 U.S.C. 102(e). The Examiner in the Office Action dated May 7, 2003 cited the Sughrue'766 patent.

2. Attached hereto is Exhibit A. Exhibit A is a collection of laboratory notebook pages prepared prior to August 25, 1999. Exhibit A evidences acts constituting conception and reduction to practice of a claimed invention. We do not wish to disclose the dates of these materials; therefore, the actual dates have been

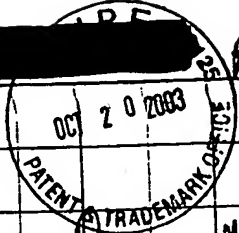
blocked out in Exhibit A. The acts evidenced by the materials of Exhibit A were performed in the United States prior to August 25, 1999.

3. The first four pages of the laboratory notebook disclose the method of making of a composition containing silica, alumina, zinc oxide, sodium silicate, and nickel. A mixture comprising alumina, silica, zinc oxide, and sodium silicate was dried, calcined, and then impregnated with nickel nitrate. The composition is then impregnated with a sodium silicate solution. The fifth and sixth pages disclose reducing the composition with hydrogen.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

Gyanesh P. Khare
Gyanesh P. Khare
October 7, 2003
Date

Attachment



30011-6
 repeat of 35161-97 with less H_2O & More Sodium Silicate

Prepared material for Spray drying using the following materials.

1. 20.0g Sodium Pyrophosphate
2. 1690 g DI Water
3. 200.0g Vista Dispal 180 - Al_2O_3
4. 471g of Celite filter Cell - Silica
5. 788g ZnO
6. 820 Sodium Silicate *

Material was no problems to Pump, a little on the thick side.

Conditions during run were:

Atomizer Press 0.3 - 0.38 Bar, operation Shu III

Percent timer 10,

Inlet Temp $320^{\circ}C$

Outlet Temp = $140-145$

Air Dampers = 5 from Closed, Pump Speed = 3.8

Used the external mix nozzle w/ 0.3 orifice

Material Spray dried Good & Made Very Good material.

Will Split material from bottom Catch (1114g) & dry only $\frac{1}{2}$ & dry & calcine the other $\frac{1}{2}$.

Made 365g in Top Catch.

Des: 38011-6

Sieve Analysis on 38011-6

Rune Sieve Analysis on 38011-6 used 50.0g of material that was D+C -50+270,

+50	- 0g
100	3.14
140	8.87
170	7.03
200	7.82
270	12.04
-270	11.04
	<hr/> 44.94

$$\begin{aligned} LBD &= 0.74 \text{ cc/g} \\ PBD &= 0.86 \\ PU &= 0.4 \end{aligned}$$

Note: Material formed a brick^{chip} after Calcination that broke apart Very Easy.

B. Cass

Impregnated 100.0g of 3801-6 w 29.71g of Nickel Nitrate that was dissolved in 22.97g of DI water. Used a PU of 0.4. This is a 6% Ni loading.
Des: 38011-6-I-6 LBD = 0.84 PBD = 0.96

See Page 40 for S.S. imp

B. Cass

Impregnated 100.0g of 38011-6 w 12% Nickel Nitrate. Dissolved 59.42g of Nickel Nitrate in 11.94g of Dist. H₂O

Des: 38011-6-I-12

Attrition Test on 5 grams of 38011-6-I-12 sieved on -100/+325 mesh

Filter end wt. 85.44

5/16" 83.90
1.54 grams

-400 mesh Fines
from remaining
material in jet cup
2.39g

2.39
1.54

3.93 = $5 \times 100 =$

78.61%

407.24
403.65

3.59

RECORDED BY B. Cass

DATE

WITNESSED BY

G. P. Khare

DATE

Impregnation of 38011-6I-6 w Sodium Silicate

Used the Sona Tek atomizer to impregnate 50.0g of 38811-6I-6 that had been heated to 300°F , with a Sodium Silicate Solution made by diluting 20ml of Sodium Silicate with 5ml of DI water.

Sodium Silicate was 9.1% Na_2O + 29.2% SiO_2 .

Dried & Calcined in a Stagnant Air oven programmed to ramp at $5^{\circ}\text{C}/\text{min}$ to 120°C & hold 2 hrs then ramp to 538°C & hold 1 hr then shut off.

Des: 38011-40

P. Case

DI Attrition Test on -40

Loaded 5.00g of -100 + 325 mesh material into Jet

Cup

Reset Air flow to Std rate of 2 L/min. Started heat on incubator.

1.15 Jet Cup Starting wt $G = 408.79\text{g}$ filter ending wt $G = 84.60$

Jet Cup Ending wt $G = 407.67$ filter Starting wt $G = 83.59$

5. 1.12 - 388

1.1g

Of the 3.88g recovered from the Jet Cup, 3.20g was +400 mesh.

DI ≈ 36

This material fluidized the same all thru out the run.

RECORDED BY B. Case

DATE

WITNESSED BY

G. P. Khare

DATE

Impregnation of 38011-40 with 24% N for total of 30% N.

Donald Engelbert

I impregnated 24 grams of 38011-40 with a solution of 14.26 grams of $N_i(NO_3)_2 \cdot 6H_2O$ and 3.27 grams of distilled H_2O .

I put material in oven and ramped the temp $3^\circ C$ a min to $150^\circ C$ and held 1 hour. I then ramped the temp ~~up~~ $3^\circ C$ a min to $635^\circ C$ and held for 1 hour.

11-3-98 Donald Engelbert

I impregnated above material (26.88 grams) with a solution of 15.97 grams of $N_i(NO_3)_2 \cdot 6H_2O$ and 3 grams of distilled H_2O .

Dried and Calcined same as above. Des 38011-74.

11-4-98 Donald Engelbert

Carison Index Attrition Test on 38011-74

I loaded 5 grams of 38011-74 sieved on -100/+325 MESH

Filter ending weight = 84.83

Jet cup ending weight = 408.30

" starting weight = 84.19

" " start " = 403.73

.64

4.57

I sieved remaining material in jet cup on 400 mesh sieve
There was .16g ^{RE} ~~remaining~~ - 400 mesh

.64

.16

.80 ÷ 5 × 100 = 16%

DI

LSM/KR tested on 58011-14

Loaded 10.00g of 38011-74 -100 +325 Mesh into RX + Started Heating unit to 700° under N₂.

11:20 RX Bed Temp = 709 Started flowing H₂

1240 Bed Temp 614 Jacket Temp 598 Start H₂ @

150cc/min and N₂ @ 150cc/min Start Gasoline.

1340 Bed Temp 639 Jacket Temp 599 Collected 1 hour sample ^{wt = 8.00}

Des 38294-58-1-1 Clear _{SI = 55}

1440 653

599 Collected 2 hour sample ^{wt = 9.63}

Des 38294-58-1-2 Clear _{SI = 70}

1540 669

599 Collected 3 hour sample ^{wt = 9.40}

Des 38294-58-1-3 Clear _{SI = 60}

Shut H₂ off Reduced N₂ to ~50cc/min Reduced temp to 300

0700 increased temp setting to 900°. Set N₂ flow rate to 240cc/min

0730 Bed temp = 900, Started Air flowing at 60cc/min

0835 Shut Air off & reduced temp setting to 700

Set H₂ rate to 300cc/min

0845 Bed temp = 707 Started H₂ at 300cc/min N₂ off

0945 Bed temp = 709 Reduced temp setting to 600°F Reset

N₂ & H₂ flow to 150cc/min

10:30 Bed temp = 621 Jacket = 599 Started H₂ & N₂ at 150cc/min each.

also Started Gasoline at 13.4cc/hr.

RECORDED BY B. G. D. 2 DATE [redacted] WITNESSED BY G. P. Khare

DATE [redacted]

- 11:30 Bed temp = 647 Jacket = 599 Collected 1 hr Sample wt = 7.80 5
Des: 38294-59-2-1 Clear 5
- 12:30 Bed temp = 682 " 599 Collected 2 hr Sample wt = 9.35g Clear 15
Des: 59-2-2
- 1:30 Bed temp = 679 " 599 Collected 3 hr Sample wt = 9.7g Clear 20
Des: 59-2-3
- 2:30 Bed temp = 679 " 599 Collected 4 hr Sample wt = 9.67 Clear 20
Des: 38294-59-2-4
- 3:30 " = 694 " 599 Collected 5 hr Sample wt = 9.20 Clear 15
Des: 38294-59-2-5
- 4:00 Shut H₂ off & Reduced temp setting to 150°C

P. Carr

- 0700 Increased temp setting to 900° Set N₂ flow rate to 240 cc/min
- 0730 Bed temp = 900 Started Air flowing at 60 cc/min
- 0800 Shut Air off & reduced Temp setting to 700
Set H₂ + ~~N₂~~ rate to 300 cc/min
- 0845 Bed temp = 709 Started H₂, N₂ off
- 0945 Reduced temp setting to 600° F. Reset H₂ & N₂
flow rates to 450 cc/min ↑
- 10:00 Bed temp = 611 Jacket = 595 Started H₂ & N₂.
Started Gasoline Pump at 13.4 cc/min

RECORDED BY B. Carr

DATE

WITNESSED BY

G. P. Khare

DATE